

PATENT
CASE 4233C3

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Donald B. Appleby et al : Group Art Unit: 1211
Serial No.: 08/360,184 : Examiner: E. White
Filed: December 20, 1994 :
For: **Polyol Polyester Synthesis**

DECLARATION UNDER 37 C.F.R. §1.608 OF HAROLD R. McPEEK

Assistant Commissioner for Patents
Washington, DC 20231

Dear Sir:

I, HAROLD R. McPEEK, declare that:

1. I was employed by the assignee of the present application, The Procter & Gamble Company, from 1969 to 1994.

2. From 1986 to 1994 I worked as an operator/technician on the sucrose polyester synthesis project in which sucrose and fatty acid methyl ester were reacted to form sucrose fatty acid esters. I was responsible for analyzing samples of reactor streams from pilot plant continuous sucrose polyester production processes according to established procedures.

3. I participated in the pilot plant continuous sucrose polyester production process designated P90117 which was conducted from January 18, 1989, to January 27, 1989. The P90117 pilot plant continuous sucrose polyester production process, including operation of the continuous sucrose polyester synthesis equipment, sample collection and data preparation, was performed under the direction and control of Mr. Scott Pearson.

4. During the P90117 pilot plant continuous sucrose polyester production process, I determined the weight percentage of unreacted sucrose in samples taken from the recirculation loop of the initial reactor, i.e., the first reactor R600, according to the established procedure described below, and I accurately recorded the determined weight percentage of unreacted sucrose in the samples on data sheets which were maintained in the pilot plant for the purpose of recording such data. The data which I accurately recorded included the date and time at which a sample was taken, the reactor recirculation loop from which the sample was taken, and the determined weight percentage of unreacted sucrose in the sample. I also initialed the recorded data for each sample which I analyzed.

5. I have examined Exhibit 9, and I confirm that Exhibit 9 comprises accurate copies of the data sheets from the P90117 pilot plan continuous sucrose production process on which I accurately recorded the date and time at which a sample was taken, the reactor recirculation loop from which the sample was taken, and the determined weight percentage of unreacted sucrose in the sample. The entries bearing my initials (HRM) indicate those samples which I analyzed as described herein and for which I recorded the data as described herein.

6. The established procedure for determining the weight percentage of unreacted sucrose in a sample during the P90117 pilot plant continuous process employed a YSI-27 Industrial Analyzer. In the analyzer, the sucrose is enzymatically converted to glucose, the glucose is enzymatically oxidized to generate hydrogen peroxide, and the hydrogen peroxide is electrochemically oxidized, yielding a response proportional to the sucrose concentration. According to the established procedure, the YSI-27 Industrial Analyzer was calibrated using standards of 1000 and 2000 mg/dl. A weighed amount of the liquid reactor sample was mixed with about 95 milliliters of warm tap water (about 160°F) and the diluted sample was stirred at a temperature of from about 150°F to about 160°F for about 5 minutes. The sample was removed from the heat source and an oil layer was allowed to rise to the surface. A portion of the aqueous layer was collected and injected into the YSI-27 analyzer. The analyzer output was read and the percentage of unreacted sucrose in the sample was determined using the following equation:

$$\text{Wt. \% Unreacted Sucrose} = \frac{\text{Instrument Read Out (mg/dl)}}{\text{Sample Weight (g)} \times 10}$$

7. During the P90117 pilot plant continuous sucrose polyester production process, I also determined the weight percentages of potassium carbonate catalyst (as carbonate) and potassium soap (as soap) in samples taken from the recirculation loop of the initial reactor stage, i.e., the first reactor R600, according to established procedures described below, and I accurately recorded the determined weight percentages of potassium carbonate catalyst and potassium soap in the samples on data sheets which were maintained in the pilot plant for the purpose of recording such data. The data which I accurately recorded included the date and time at which a sample was taken, the reactor recirculation loop from which the sample was taken, and the

determined weight percentages of potassium carbonate catalyst and potassium soap in the sample. I also initialed the entry for each sample which I analyzed.

8. I have examined Exhibit 7, and I confirm that Exhibit 7 comprises accurate copies of the data sheets from the P90117 pilot plant continuous sucrose polyester process on which I accurately recorded the date and time at which a sample was taken, the reactor recirculation loop from which the sample was taken, and the determined weight percentages of potassium carbonate catalyst (as carbonate) and potassium soap (as soap) in the sample. The entries bearing my initials (HRM) indicate those samples which I analyzed as described herein and for which I recorded the data as described herein.

9. The established procedure for determining the weight percentages of potassium carbonate catalyst (as carbonate) and potassium soap (as soap) in a sample during the P90117 pilot plant continuous process comprised titration with a 0.1 Normal HCl solution utilizing a Kyoto At-118 microprocessor-controlled automatic titrator. A weighed reactor sample was mixed with a neutral solution comprising equal parts of isopropanol and water. The sample mixture was heated with stirring until the temperature reached about 100°F and then was heated for about 2 minutes at about 100°F or until the sample dispersed completely. The sample mixture was placed on the titrator and was automatically titrated until the pH reached 3 or below. The automatic titration indicated that there were two equivalence points. The first equivalence point corresponded to the stronger base, the potassium carbonate catalyst (as carbonate), and second equivalence point corresponded to the weaker base, the potassium soap (as soap). The

weight percentages of potassium carbonate catalyst (% Carbonate) and potassium soap (% Soap) in the sample were calculated from the titration volumes needed to reach the two equivalence points, respectively, using the following equations:

$$\% \text{ Carbonate} = \frac{\text{EP1 (Vol)} \times \text{Normality HCl} \times 13.8}{\text{Sample Wt (g)}}$$

$$\% \text{ Soap} = \frac{[\text{EP2 (Vol)} - 2 \times \text{EP1 (Vol)}] \times \text{Normality HCl} \times 32.0}{\text{Sample Wt (g)}}$$

wherein EP1 was the volume of the HCl solution need to reach the first equivalence point, EP2 was the volume of the HCl solution need to reach the second equivalence point, and Normality HCl was the Normality of the HCl titration solution, as set forth on the third page of Exhibit 7.

10. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the present application or any patent issued thereon.

Respectfully submitted,

By: _____

Harold R. McPeek

Date: 2-21-99

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